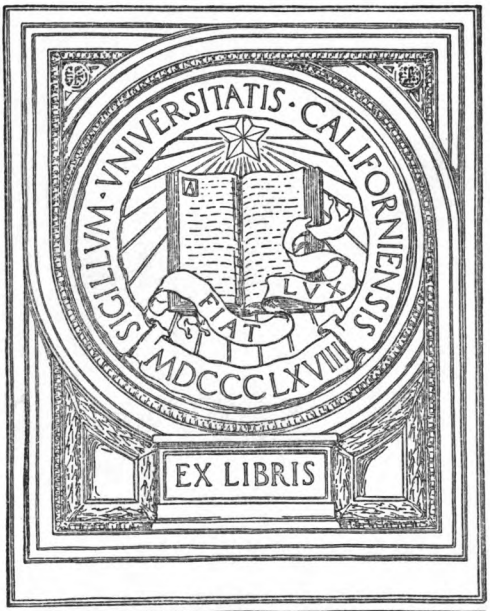

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THE PASSAGE OF HYDROGEN THROUGH QUARTZ GLASS

BY

J. B. JOHNSON and R. C. BURT

MEASUREMENTS ON THE FLOW OF HYDROGEN THROUGH QUARTZ GLASS BETWEEN 300° C. AND 800° C. FLOW BEGINS NEAR 300° C. INCREASING RAPIDLY WITH TEMP. SOME MEASUREMENTS MADE WITH NITROGEN AND ARGON

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THE PASSAGE OF HYDROGEN THROUGH QUARTZ GLASS

By J. B. Johnson and R. C. Burt

SYNOPSIS

The *Rate of Flow of Hydrogen Through Quartz Glass* has been measured over the range of 300°C. to 900°C. Some measurements were also made with *Nitrogen and Argon*. A perceptible diffusion starts with hydrogen at about 300° C. and with the nitrogen at 600° C., and then in each case increases rapidly with the temperature.

A brief discussion is given on the *Possible Nature of the Flow* of gases through fused silica.

The heat resistive property of fused quartz has made this material valuable for the construction of many kinds of scientific apparatus. In some cases, however, its usefulness has been limited by the porosity to various gases at higher temperatures. O. W. Richardson[1] found that hydrogen and helium, and to a less extent neon, diffused through a quartz glass tube at 800°–1200° C. The rate of the diffusion has been measured when the pressure on both sides of the material was fairly high,[2] but the results thus obtained cannot safely be applied to high vacuum apparatus. The increased importance of electronic discharge tubes of larger power opens a new field for the use of fused silica, provided the material is used with proper regard for its limitation. The experiments to be described were therefore done to get more definite knowledge of the behavior of this material under conditions of high vacuum and high temperature. While the flow of hydrogen was studied more fully, some measurements were also made with nitrogen and argon.

The method used in making the measurements was to observe the pressure rise in an evacuated silica glass tube around which the gas flowed. A diagram of the apparatus is shown in Fig. 1. The furnace was an iron pipe around which was wound a heater of resistance wire and a covering of asbestos. A thermocouple

[1] Phil. Mag., *22*, p. 704; 1911.

[2] E. C. Mayer, Phys. Rev., *6*, p. 283, 1915; H. Wüstner, Ann. d. Phys., *46*, p. 1095, 1915.

placed near the center of the furnace was used to measure the temperature. The gas from a commercial tank was passed through the furnace at a slow steady rate at atmospheric pressure. The quartz glass tube Q was placed axially in the furnace and was connected to a vacuum pump, a McLeod gauge and a volume bulb V, the total volume of the system being about 1500 cc. A side tube T with a volume of about 3 cc could be immersed in liquid air for freezing vapors out of the system. The difference in level of the capillary mercury columns of the gage at the time of taking a pressure reading was kept less than the vapor pressure of water. No condensed water, therefore, existed in the closed capillary, so that when the side tube was not cooled the gauge indicated

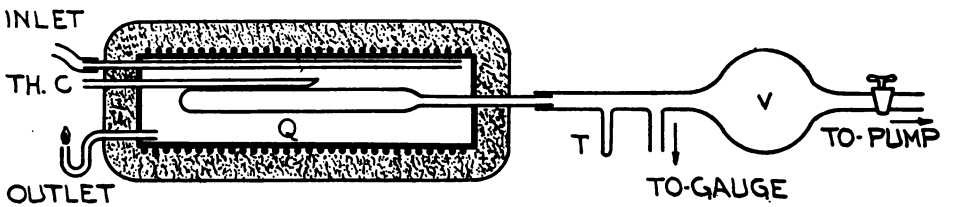

FIG. 1. Apparatus

approximately the total pressure, including that of water vapor. After the tube was pumped out the connection to the pump was closed during a run and the pressure read at short intervals while the temperature was kept nearly constant.

The materials tested were furnished by the Thermal Syndicate, Ltd., and were in the form of tubes 35 cm long, 1.5 cm in outside diameter and 1.5 mm wall thickness. Rough tests on several opaque varieties of fused silica, made from quartz sand, showed that these are unsuited for high vacuum apparatus even in the air at room temperature. The lowest rate of pressure increase observed was, for one of the tubes, about .001 mm of air per hour at room temperature and other tubes gave as high as ten times this value under the same conditions. The final work was therefore confined to the clear fused silica made from quartz crystals.

After the clear silica tubes had been heated up to drive off the vapors on the inner surfaces, no leakage of air or hydrogen at room temperatures was detected in as long a time as two weeks at a

pressure of less than .001 mm. Upon again heating the tube a measurable leak of hydrogen started first at about 300° C, and then increased rapidly with the temperature. Fig. 2 shows a summary of the results obtained with three tubes. R is the rate in cc per hour at which gas leaked into the tube per unit area, reduced to one mm pressure and room temperature, corrections being made for the temperature of the quartz and of the tube T.

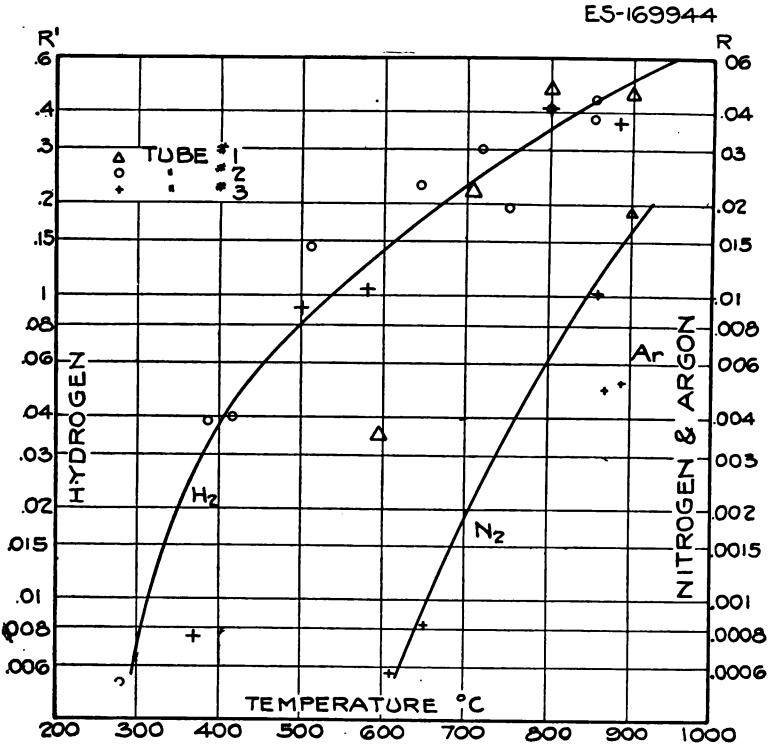

FIG. 2. Rate of Flow of Hydrogen, Nitrogen and Argon

The tubes were not quite of the same average thickness and the results have been reduced to correspond to a wall thickness of 1.5 mm, on the assumption that the leak is inversely proportional to the thickness. The runs with nitrogen and with argon are also shown in this figure. At 400° and 500° no leak of nitrogen as large as 5×10^{-4} mm was detected in 24 hours.

The result of changing from one gas to another is shown in Fig. 3, which makes clearer the difference between leakage of hydrogen and the heavier gases. The curve at the top of the figure gives the temperature of the oven during the course of the experiment. The upper pressure curve gives the total pressure in the system, while the lower curve gives the pressure of permanent gas measured when the small side tube was immersed in liquid air. The difference between the two curves represents the pressure of condensible vapors, about 90% of which was water vapor as was shown by exposing the gas to a small amount of

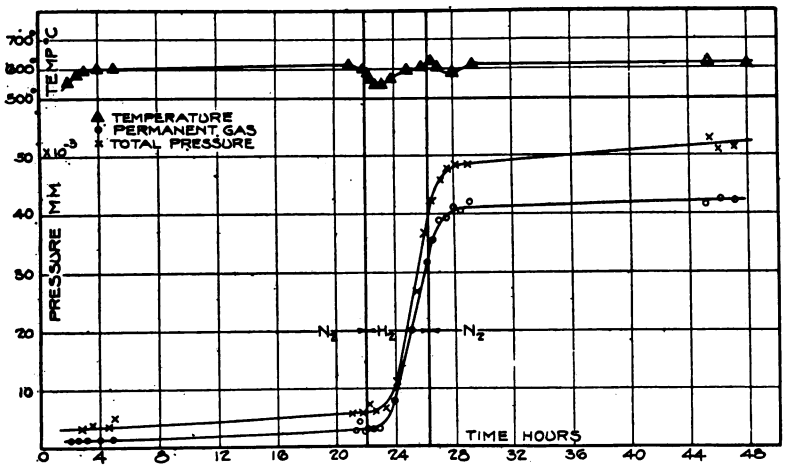

FIG. 3. Change of Gas Surrounding the Tube

phosphorus pentoxide. The presence of this condensible gas suggests the possibility that the rise in pressure was caused by gases continually given off from the walls of the tube rather than by a leakage through the walls, but the changes in slope show conclusively that there is a leakage of hydrogen at least. The constancy with time indicates that the lower rate of increase of pressure is caused by leakage of nitrogen as has been assumed and only to a small extent by release of gases from the inner surface of the tube.

The results of these experiments, though incomplete, justify a brief discussion of the nature of the flow of gas through fused silica. It seems improbable that a clear, dense substance such as

quartz glass is porous to gases in the sense that chalk is porous. It is equally improbable that in this inert material there is a chemical diffusion as in rubber. An assumption which might readily be made is that there is a flow of gas through very fine holes or tubes in the material. This view gains apparent support from the fact that even clear fused silica is not quite free from fine striations, caused by drawn out air bubbles, a condition which is so obviously present in the satin finish material. According to the kinetic theory, however, the volume of gas passing through a tube when the diameter of the tube is small compared with the mean free path of the gas molecules is inversely proportional to the three-halves power of the molecular weight and directly proportional to the square root of the absolute temperature. When the experimental rates for the three gases are compared, their ratios fall within the correct range for the molecular weight relation, but the increase of rate with temperature is very much greater than that shown by flow through tubes and apertures, and indeed varies as the third or higher power of the temperature, so that we must look for some other explanation than simple flow along capillary tubes. The transfusion begins at the temperature at which structural changes are known to occur in crystalline silica, and this fact suggests that the passage of the gas may accompany a modification in the structure of the non-crystalline material.

The data enable us to judge what to expect with tubes and bulbs of ordinary dimensions. The curves show that at the same outside pressure hydrogen passes through the walls about 100 times faster than nitrogen or argon. Under normal conditions, however, the hydrogen content of the atmosphere is small, about four parts in 100,000 by volume, so that in air the rate of admission of nitrogen should be of the order of 250 times larger than that of hydrogen. To take a concrete example which may be met with in practice, we can probably say that a well evacuated bulb of one liter capacity can be kept in the air at 400° C for one hundred hours before the pressure reaches 10^{-4} mm, and the transfused gas will be largely nitrogen.

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